

## Thermal and mechanical characterization of polypropylene composite membrane doped with TiO<sub>2</sub> nanoparticles

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The HVAC (Heating, Ventilating, and Air Conditioning) industry offers many opportunities for membrane-based gas separation technologies. In this study, a hollow polypropylene (poly-P) fibre membrane loaded with nanoparticles was created to dry out the indoor air. The mechanical and thermal stability of polypropylene loaded nanoparticles (TiO<sub>2</sub>/poly-P) usually improve with increasing titanium concentration. The example of poly-P with a 4% volume concentration of TiO<sub>2</sub> nanoparticles in a polypropylene matrix shows the highest improvement in thermal stability. Atomic force and scanning electron microscopy were used to examine the nanocomposites' structure, and the results showed a correlation between the change in the thermal and mechanical characteristics and the change in TiO<sub>2</sub>/poly-P content. According to AFM investigations, when titanium nanoparticles are added to poly-P, the supramolecular structure is altered and an ordered structure is created. In comparison to 2% TiO<sub>2</sub> doped poly-P nanocomposites, films containing 4% TiO<sub>2</sub> demonstrated a more effective immediate moisture retention capacity, according to moisture absorption analyses. This study offers a fresh viewpoint for enhancing the poly-P composite membrane's ability to dehumidify the air.

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### 1. Introduction

The use of membrane-based technology for the separation of gases has attracted a lot of attention and shown a lot of promise because it consumes less energy, is simpler to configure, requires fewer maintenance components, and is more favorable to the environment[1], [2]. The market that deals with heating, ventilation, and air conditioning (HVAC) is becoming more interested in one of the many uses of the membrane method known as the dehumidification of indoor air. However, to further develop typical polymeric membranes, a trade-off between permeability and selectivity has needed to be explored[3], [4]. Numerous researchers have taken an interest in the alteration of the membrane surface to improve the membrane's characteristics by incorporating of nanoparticles into the membrane layer[5], [6]. In particular, the incorporation of nanoparticles into the membrane material results in a significant improvement in both the membrane's wettability and specificity. It is a work of urgent importance to create composites with distributed metallic phases with dimensions of the metal nanoparticles of nanoscale size since these nanoparticles have a variety of newly discovered features owing to their excess surface energy[7]. It should be mentioned that there are not many publications concerning the effect that the kinds of nanoparticle additions have on the dehumidification performance of composite membranes as well as the settings under which they are included. We report on the thermal and mechanical characteristics of TiO<sub>2</sub>/poly-P polymer nanocomposites in this research. These nanocomposites were created by introducing TiO<sub>2</sub> nanoparticles into polypropylene (poly-P). The moisture absorption test was carried out to investigate the capability of the manufactured composite membrane to remove moisture from the air.

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## 2. Experimental methods

### 2.1. Synthesis of TiO<sub>2</sub> nanoparticles

TiO<sub>2</sub> nanopowder was produced by following a process that was previously reported[8]. After stirring magnetically for a total of two hours, about 4 ml of titanium (IV) isopropoxide (Himedia) was dissolved in a volume of 50 ml of double-distilled water. After that, 19.18 grams of NaOH were added to create a solution of 5 M, which was then autoclaved at a temperature of 200°C for 24 hours. The resultant powder was mixed with HCl (37.96%) for a period of thirty minutes to remove any surplus of sodium ions and bring the pH to 2. After that, the powder of titanium dioxide was extracted and washed many times in double-distilled water until it reached a pH of 7. After the preceding step was finished, the powder was dried in an air environment for six hours at a temperature of 100°C.

### 2.2. Preparation of TiO<sub>2</sub> polymer composite membrane

The following are the procedures that were taken to create polymer nanocomposite materials based on poly-P+TiO<sub>2</sub>: At a temperature of 120°C, an isotactic polypropylene powder manufactured by Sigma-Aldrich and having particles ranging in size from 0.5 to 1.0 micron was dissolved in toluene. To get a homogenous mixture, the nanoparticles that had been previously made were introduced into the polymer solution, and the combination was agitated for a total of two hours. The samples were vacuum dried for one day to get rid of any remaining solvent. After subjecting these samples to a hot-pressing process at the polypropylene melting temperature while maintaining a pressure of 10 MPa, several thin films with varying film thicknesses were produced. As a result, nanocomposites with 0, 2, and 4% of the TiO<sub>2</sub> filler were manufactured. After being subjected to hot pressing, the films were cooled in water at a rate of 200 K per minute[9].

### 2.3. Characterization

At ambient temperature, the XRD diffractograms of nanocomposites were analyzed using a diffractometer called a Rigaku Mini Flex 600s. This instrument used an X-ray tube with a copper anode to produce Cu-K $\alpha$  radiation at a setting of 30 kV and mA. Scanning electron microscopy was used to investigate the nanoparticles of titanium's dispersion inside the polymer matrix (SEM, Jeol JSM-7600 F). A thermogravimetric analyzer (TGA) Model Perkin Elmer Pyris was used for the process of performing the thermogravimetric analysis on the samples. In the presence of nitrogen, samples of nanocomposites were subjected to a temperature increase from 25°C to 550°C at a rate of 20°C per minute. Using atomic force microscopy equipped with a Model-PicoSPM-2100, the morphology of the nanocomposites was analyzed (USA). For the scan, specialized silicon cantilevers were manufactured using a plasma etching approach. These cantilevers had a radius of curvature of the needle that was 20 nm, and their resonance frequency was between 1 and 5 Hz. The scan size was 5 microns on each side. The measurements were carried out using a semi-contact microscopy mode in air, with a fixed needle change of the cantilever oscillation amplitude. This enables the surface topography to be determined. Both the speed of the scanning and the number of lines that are being scanned across the picture are 256 and 1,969 Hz, respectively[10].

### 2.4. Moisture absorption test

The samples used in the test were 1 cm  $\times$  3 cm and were dried in an oven until they reached a consistent weight. The initial weight, denoted by ( $W_I$ ), and the final weight, denoted by ( $W_F$ ), are as follows: The moisture absorption was performed in a chamber with a relative humidity of 99% and a temperature of 25°C. The final weight ( $W_F$ ) of the sample is determined by reweighing it after every 30 minutes. According to the findings of a prior investigation, the percentage of moisture absorption was computed using the equation shown below[11].

$$\% \text{ of Moisture Absorption} = \frac{W_F - W_I}{W_I} \times 100 \quad (1)$$

### 3. Results and discussion

The diffraction patterns of nanocomposites based on poly-P that include varying amounts of titanium nanoparticles in their volume are shown in Figure 1. The primary peaks of 25,27 (101), 37,91 (004), and 47,90 (200) correspond to the titanium nanoparticles, as shown by the XRD diffraction patterns. For this particular design, the PDF number 21-1272 which corresponds to titanium may be used to index all of the lines[12]. As can be observed, an increase in the amount of titanium nanoparticles present in the polymer results in an increase in the nanocomposites' degree of crystallinity[13].

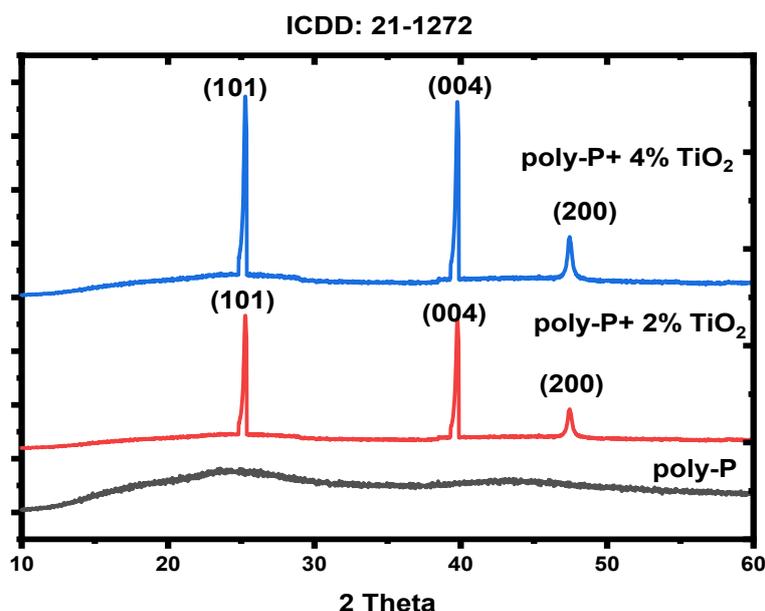


Fig. 1. X-ray diffraction patterns of poly-P+TiO<sub>2</sub> nanocomposites with different percentages of TiO<sub>2</sub>.

SEM pictures of nanocomposites made of poly-P+TiO<sub>2</sub> are shown in Figure 2. The increase in the volume of TiO<sub>2</sub> nanoparticles in the polymer matrix, as shown in figure 2, resulted in an increase in the quantity and size of nanoparticle clusters in the poly-P matrix[14]. TiO<sub>2</sub> nanoparticles in the matrix have a size range of 20–85 nm at 2% filler content and 35–80 nm at 4% filler content (Figure 3).

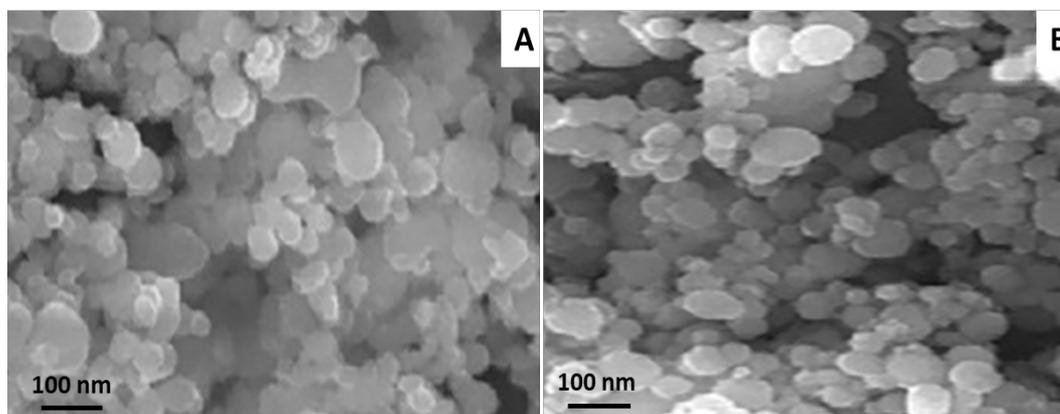


Fig. 2A. SEM images of poly-P +2% TiO<sub>2</sub>; 2B. poly-P + 4% TiO<sub>2</sub>.

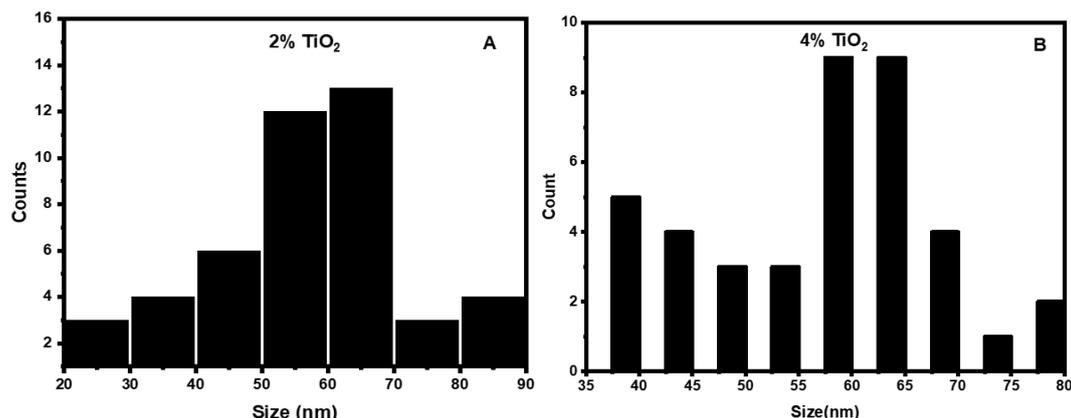


Fig. 3. Histogram size distribution (nm) A) poly-P + 2% TiO<sub>2</sub>; B) poly-P + 4% TiO<sub>2</sub>.

AFM pictures of poly-P+TiO<sub>2</sub> based nanocomposites with varying titanium volume contents are shown in Fig. 4. As can be seen from figure 4, the creation of a more ordered structure was caused by the inclusion of titanium nanoparticles up to 4% of volume content in the polypropylene matrix. Thus, adding 2% Ti to the polymer caused the titanium nanoparticles to distribute rather evenly, but nanocomposite with 4% Ti exhibited a more flawless structure. As the titanium nanoparticle concentration in the polymer increased further, it is clear from the AFM picture that this caused agglomeration processes in the matrix[15]. This closely connects to the average surface roughness values for nanocomposites. The surface roughness histograms of the nanocomposites made of poly-P+TiO<sub>2</sub> are shown in Figure 5. Thus, the nanocomposite poly-P+2%TiO<sub>2</sub> and poly-P+4%TiO<sub>2</sub> had an average surface roughness of 35–65 nm and 70–90 nm, respectively. This leads one to the conclusion that nanocomposites have a more perfect supramolecular structure at low nanoparticle concentrations than they do at high nanoparticle concentrations in the polymer matrix[16].

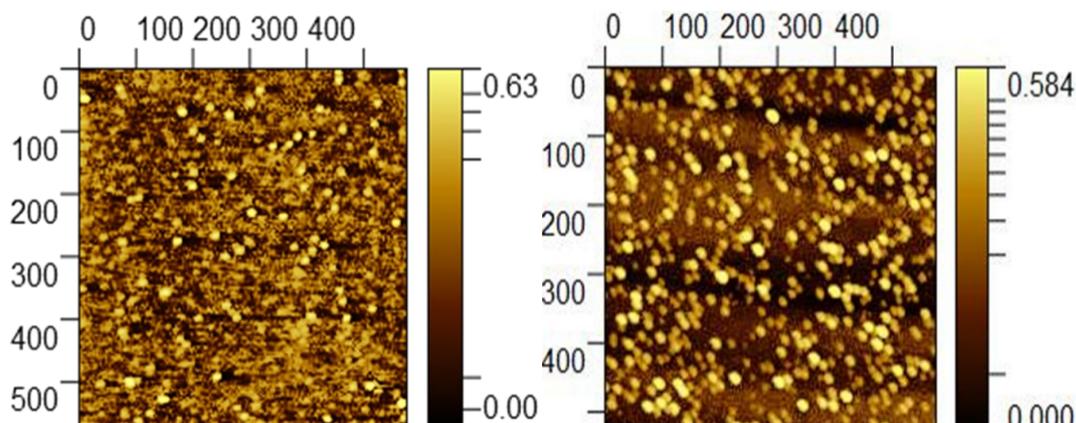


Fig. 4. AFM images of poly-P+TiO<sub>2</sub> nanocomposites a) poly-P + 2% TiO<sub>2</sub>; b) poly-P + 4% TiO<sub>2</sub>.

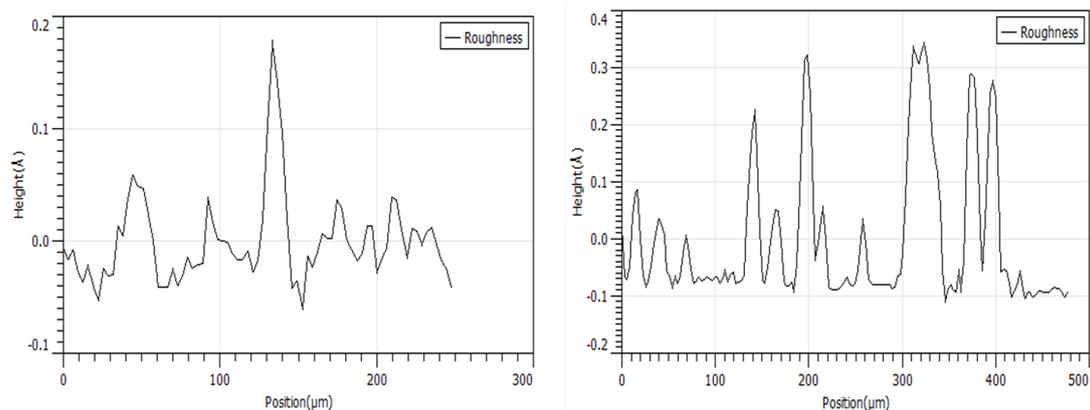


Fig. 5. Poly-P+TiO<sub>2</sub> nanocomposites roughness histogram a) poly-P +2% TiO<sub>2</sub>; b) poly-P + 4% TiO<sub>2</sub>.

To ascertain the thermal stability of poly-P+TiO<sub>2</sub> nanocomposites, thermogravimetric analysis (TGA) of nanocomposites was conducted. The TGA curves of poly-P+TiO<sub>2</sub> nanocomposites are shown in Fig. 6. The thermo-oxidative degradation of polymer nanocomposites happens in a single step, as can be shown in Figure 6. At 348, 240C, poly-P starts to break down, which is measured as a 1% weight loss and continues to do so up to 472, 330C. As can be seen from the TGA curve, the weight remains constant after reaching 472, 330. The breakdown peak migrated to higher temperatures (up to 377, 330) when 2% TiO<sub>2</sub> nanoparticles content was added to the polypropylene[17]. The weight of the nanocomposite keeps decreasing until it reaches 474,530 and then stays the same. The breakdown temperature changes back to lower temperatures as the iron concentration in the polypropylene matrix continues to rise. Therefore, 321,730° is the breakdown temperature for nanocomposites containing 4% TiO<sub>2</sub>. As can be seen, adding TiO<sub>2</sub> nanoparticles to the polypropylene matrix has a significant impact on the prepared nanocomposite's thermal resistance. TiO<sub>2</sub> nanoparticle distribution within polypropylene also increases the material's thermal stability, and this effect lasts up to 2,4% volume content of TiO<sub>2</sub> nanoparticles. The thermal stability of nanocomposites declines as iron nanoparticle loading in the polymer matrix continues to rise. As a result, the breakdown temperature of the nanocomposite moves to higher temperatures (up to 29<sup>0</sup>C) than the pure polymer at a 2,4% loading of TiO<sub>2</sub> nanoparticles[18].

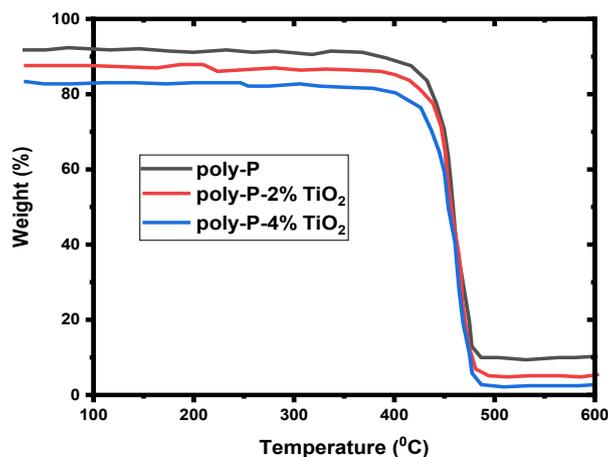


Fig. 6. TGA of poly-P+TiO<sub>2</sub> nanocomposites.

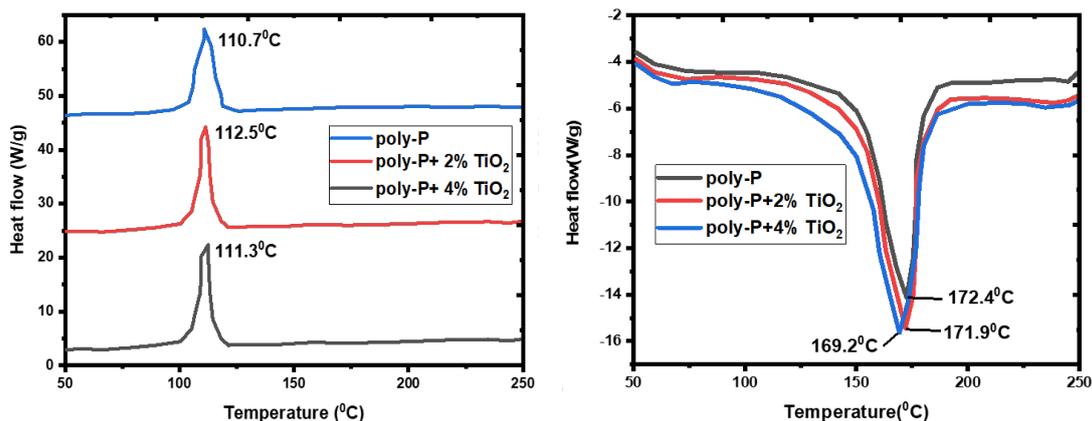


Fig. 7a. DSC of poly-P+TiO<sub>2</sub> nanocomposites b. DSC showing melting temperatures poly-P +2% TiO<sub>2</sub> and poly-P + 4% TiO<sub>2</sub>.

Based on the differential scanning calorimetric (DSC) analytical technique, information on thermal phase transition in poly-P+TiO<sub>2</sub> nanocomposites was acquired (Fig. 7a and 7b). The crystallization temperature of nanocomposites rises with a shift towards higher temperatures as the TiO<sub>2</sub> nanoparticle loading in the polymer matrix increases. The literary study makes it abundantly evident that only powerful structural elements can have an impact on the temperature at which nanocomposites crystallize. Thus, it was noted that TiO<sub>2</sub> nanoparticles operate as active structural constituents and may move the crystallization peak to higher temperatures[19]. Additionally, it has been shown that adding TiO<sub>2</sub> nanoparticles to poly-P polymer matrices might lower their melting point. Table 1 lists the calculated degree of crystallinity for poly-P+TiO<sub>2</sub> nanocomposites. The formula to determine the degree of crystallinity using the crystalline polypropylene's fusion enthalpy is 166 J/g;

$$\text{Crystallinity (\%)} = \frac{\Delta H_{\text{Sample}}}{\Delta H_{\text{Sample}}^0} \quad (2)$$

Mechanical durability of the power based on  $\log(\sigma)$  has been investigated in order to forecast the expiry date of nanocomposites and interfacial contacts of poly-P+TiO<sub>2</sub>, as well as for the study of kinetic processes happening in these systems. Testing the mechanical capabilities of apparatus that enable the experimenter to guarantee the stability of the rupture stress. The impact of TiO<sub>2</sub> nanoparticle concentration on poly-P+TiO<sub>2</sub> nanocomposite's mechanical durability. Figure 8 depicts the relationship between the mechanical stress and the logarithm of mechanical durability,  $\log(\sigma)$ . It was discovered that the following regularity governs how mechanical durability changes[20]. Specifically, the equation

$$\tau = \tau_0 \cdot e^{\frac{U_0 - \gamma\sigma}{kT}} \quad (3)$$

where,

$\tau_0$ - Constant

$U_0$ - Thermal degradation activation energy

$\gamma$ - Structural sensitivity factor

$\sigma$ - Mechanical strength of the material

Table 1 shows that the degree of crystallinity rises, then falls, up to a concentration of 2% TiO<sub>2</sub> nanoparticles.

Has the relationship between the concentration of TiO<sub>2</sub> nanoparticles and the mechanical characteristics of poly-P+TiO<sub>2</sub> nanocomposites been studied (Fig. 8). It was noted that the mechanical strength of nanocomposites initially rises to 2% of the volume content of TiO<sub>2</sub> nanoparticles, and subsequently decreases with additional increases in nanoparticle concentration[21]. It is essential to emphasize that the degree of interaction between the polymer

matrix and nanoparticles relies on the main phase concentration, the size of the nanoparticles, and the method used to generate the nanocomposites.

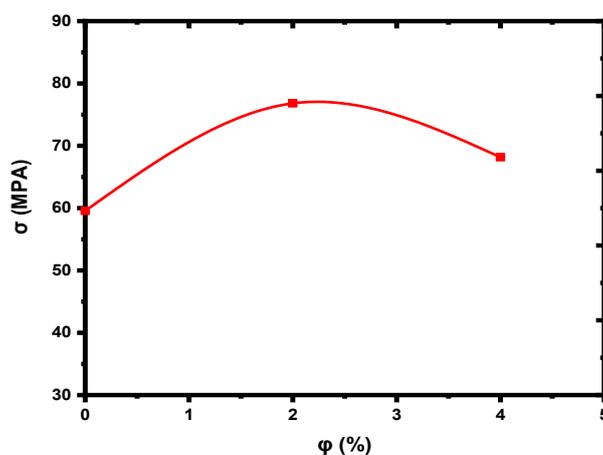


Fig. 8. Dependence of mechanical strength of poly-P+TiO<sub>2</sub> nanocomposites based on the weight content of TiO<sub>2</sub>.

A border layer or interphase with different characteristics from the matrix is formed at the interface between the filler and the matrix[22]. While nanoparticle structuring activities occurred in the nanocomposite's boundary layer, the crystal polymer structure was created. The thermodynamic conditions for the crystallinity of the molecular chains on the boundary layer are thought to have improved as a result of the structural activities of nanoparticles. Therefore, the researchers investigated the structural, thermal, and mechanical characteristics of poly-P+TiO<sub>2</sub> nanocomposites. The supramolecular structure of the polymer, the polymer's polarity, and the volume content of the nanoparticles have all been shown to have a significant impact on the characteristics of polymer nanocomposites[23].

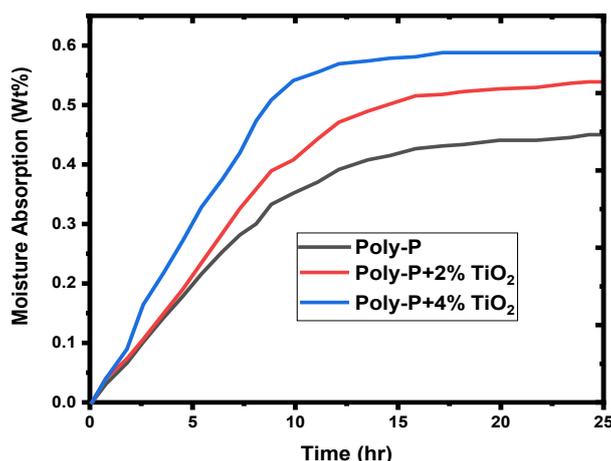


Fig. 9. Moisture absorption characteristics of poly-P+TiO<sub>2</sub> nanocomposites based on the weight content of TiO<sub>2</sub>.

It has been shown that supramolecular structures with higher order are generated in polymers with low TiO<sub>2</sub> concentrations in the matrix as opposed to those with high TiO<sub>2</sub> concentrations. While TiO<sub>2</sub> nanoparticles act as fillers, the subsequent increase in TiO<sub>2</sub> volume content causes the creation of a more ordered structure and results in a somewhat orderly dispersion of TiO<sub>2</sub> nanoparticles within the polymer matrix. According to definitions, modest

amounts of iron nanoparticles serve as structural elements in a polymer matrix; nevertheless, when the quantity of TiO<sub>2</sub> in nanocomposites increases, the physical structure of polymers is destroyed. As a result of the creation of a more ordered structure at lower nanoparticle concentrations, the characteristics of these nanocomposites are improved[24,25]. Figure 9 displays the findings of water absorption in an ambient chamber at 23°C for poly-P-TiO<sub>2</sub> nanocomposites. As was seen, the adsorption rate is first high and then gradually becomes constant owing to sample saturation. The mass of water adsorbed grows linearly with time, then achieves equilibrium owing to saturation when Fick's diffusion behaviour was plotted between graphs. The use of nanoparticles as fillers increases the water absorption capacity, as shown by the superior absorption behaviour of nanocomposites containing 4% TiO<sub>2</sub> as compared to 2% TiO<sub>2</sub>. Poly-P, Poly-P+2% TiO<sub>2</sub>, and Poly-P+2% TiO<sub>2</sub> nanocomposites were reported to have an absorption capacity of 0.489 (wt%) and 0.578 (wt%), respectively. It was assumed that the absorption rate of nanocomposites increased as TiO<sub>2</sub> concentrations improved.

#### 4. Conclusion

In this study, isotactic polypropylene and TiO<sub>2</sub> nanoparticle-based nanocomposites were examined for their thermal and mechanical characteristics. TiO<sub>2</sub> nanoparticles were initially produced using a soft chemical technique and added to the poly-P matrix. XRD is used to identify TiO<sub>2</sub> in poly-P, and it improves the mechanical and thermal stability of nanocomposites. TiO<sub>2</sub> concentration in polypropylene nanoparticles normally rises along with the thermal stability and mechanical qualities. TiO<sub>2</sub> nanoparticles included in the polypropylene matrix at a volume level of 2% exhibit the greatest increase in heat stability. By using atomic force and scanning electron microscopy to examine the structure of nanocomposites, it was possible to determine how the amount of TiO<sub>2</sub> in the composites affected their thermal and mechanical characteristics. According to AFM investigations, when TiO<sub>2</sub> nanoparticles are added to polypropylene, the material's supramolecular structure changes, and an ordered structure is created. The structure of a polymer nanocomposite becomes more uneven when the TiO<sub>2</sub> nanoparticle loading is increased. Thus, for titanium nanoparticle contents of 2% and 4% in polypropylene nanocomposites, the roughness has a value of 40–60 nm. Accordingly, it has been discovered that increasing the quantity of TiO<sub>2</sub> nanoparticles damages the physical structure of polypropylene and that the concentration of 2% to 4% plays a key function in the adjustable structures. Further, the improved absorption behaviour of nanocomposites containing 4% TiO<sub>2</sub> over 2% TiO<sub>2</sub> demonstrates that the use of nanoparticles as fillers boosts the water absorption capacity. Based on the aforementioned results, it was suggested that TiO<sub>2</sub> nanoparticles filled with Poly-P can be used as fillers for prospective air dehumidification applications.

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